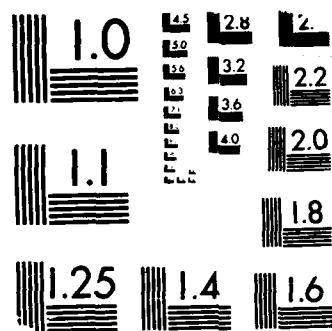


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Correlations of Norbornenyl Crosslinked Polyimide Resin Structures With Resin Thermo-Oxidative Stability, Resin Glass Transition Temperature and Composite Initial Mechanical Properties

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Prepared for the
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Resin Thermo-Oxidative Stability, Resin Glass Transition Temperature
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As part^{1,2} of a continuation of a larger investigation directed toward understanding the high temperature degradation of addition cured polymers, seventy norbornenyl cured polyimide resins were processed as neat resin discs and their long-term thermo-oxidative stability at 316°C, and glass transition temperatures (Tg) were determined after curing and postcuring at 316°C. The resin variables investigated in this study were systematic changes in both the monomeric composition and in the molecular distance between the norbornenyl crosslinks. From the resin compositions that exhibited satisfactory processing flow, thirty different unidirectional Celion 6000 graphite fiber composites were fabricated and characterized for composite initial mechanical properties and, for later portions of the larger study, long-term composite thermo-oxidative stability and composite mechanical property retention at 316°C. The purpose of this portion of the larger study was threefold. The first purpose was to determine if any relationships existed between the rate of resin weight loss per unit surface area and the monomer combinations used. The second purpose was to determine if any relationships existed between Tg and changing the distance and/or monomer variation between the norbornenyl crosslink sites. The third purpose was to determine if any relationships existed between any molecular structural modification/Tg relationship observed and the composite initial mechanical strengths obtained. What was observed for the thermo-oxidative stability portion¹ of the study was the 316°C weight loss always decreases with decreasing aliphatic content. However, more importantly, the 316°C weight loss decreases in direct correlation to increasing benzylic/aliphatic stoichiometry over a wide range of resin compositions when the norbornenyl resins contain benzylic type groups, as shown in the figures in reference 1. In contrast, the 316°C weight loss for non-benzylic/norbornenyl resins only competes with the benzylic/norbornenyl resins when the resins are based on 6F dianhydride (diacid-diester, HFDE) used with no diamine connecting group (as in paraphenylene-diamine, PPDA). All the other non-benzylic/norbornenyl resins exhibit greater weight losses than either the benzylic type/norbornenyl resins or norbornenyl resins based on very stable monomer combinations as in HFDE/PPDA. What² was observed for the Tg and composite initial mechanical property portion² of the study was an almost linear correlation between Tg and molecular distance between the crosslinks, as shown in the figures in reference 2. An attempt was made to correlate Tg with composite initial mechanical properties (flexural strength and interlaminar shear strength). However, the scatter in mechanical strength data prevented obtaining as clear a correlation as was observed between Tg and changing the molecular distance between the crosslinks. Instead, only a range of composite mechanical properties was obtained at each test temperature studied (room temperature, 288 and 316°C) as shown below and in the figures in reference 2. Perhaps more importantly, what did become apparent during the

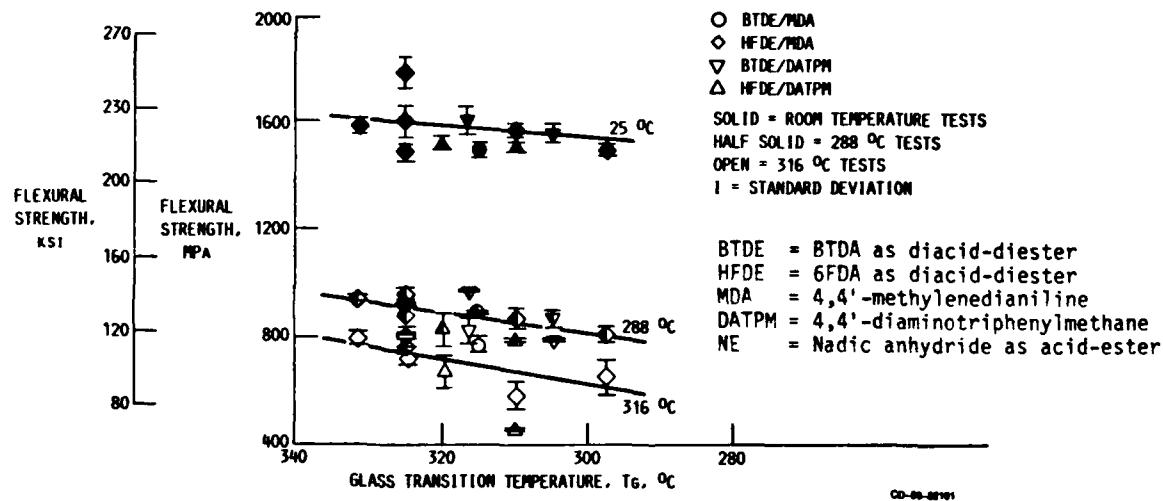
correlation study² was (a) that polymerization of monomeric reactant (PMR) methodology could be used to prepare composites from resins that contain a wide variety of monomer modifications, and (b) that these composites almost invariably provided satisfactory initial mechanical properties as long as the PMR resins formulated exhibited satisfactory processing flow.

References

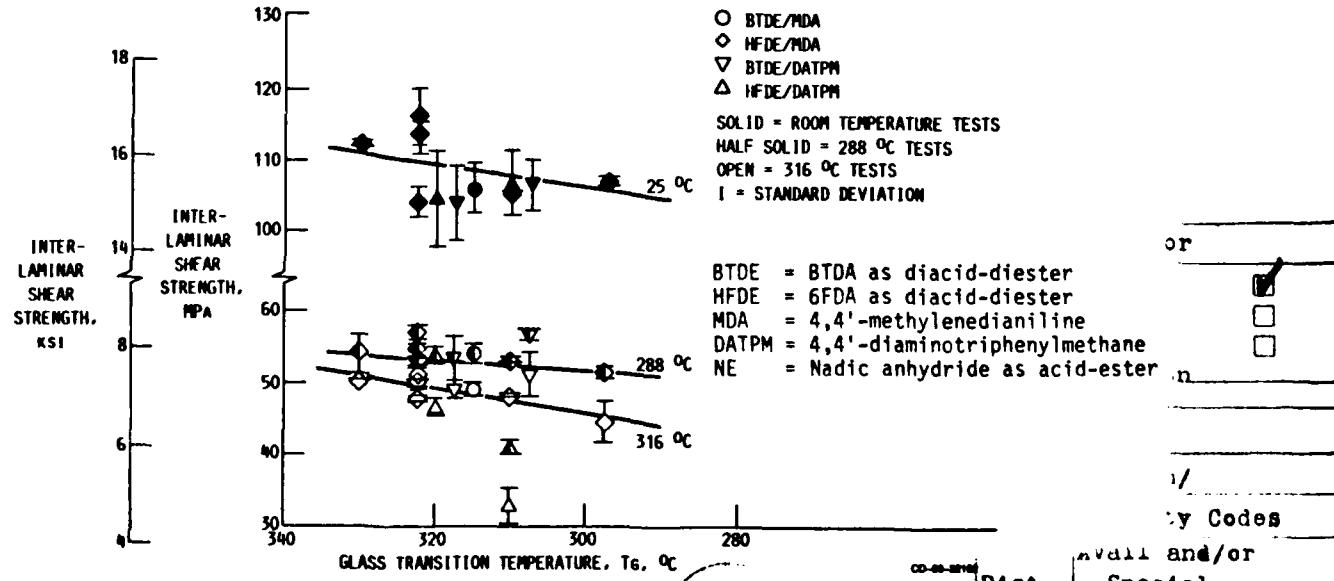
1. Alston, William B., "Structure-to-Property Relationships in Addition Cured Polymers. I. - Thermo-oxidative Stability of Norbornenyl Cured Polyimide Resins," American Chemical Society Polymer Preprints, Vol. 27, No. 2, September 1986, pp. 410-411. Also AVSCOM TR 86-C-35.
2. Alston, William B., "Structure-to-Property Relationships in Addition Cured Polymers. II. - Resin Tg and Composite Initial Mechanical Properties of Norbornenyl Cured Polyimide Resins," Proceedings of 18th International SAMPE Technical Conference, Seattle, WA, October 1986, pp. 1006-1014. Also AVSCOM TR 86-C-22, NASA TM 88794.

Tg - RESIN STRUCTURE/COMPOSITE MECHANICAL PROPERTY CORRELATION

FLEXURAL STRENGTH OF ALL BTDE AND HFDE/MDA AND DATPM/NE COMPOSITES



INTERLAMINAR SHEAR STRENGTH OF ALL BTDE AND HFDE/MDA AND DATPM/NE COMPOSITES



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16. Abstract PMR (polymerization of monomeric reactants) methodology was used to prepare seventy different polyimide oligomeric resins and thirty different unidirectional graphite fiber/polyimide composites. Monomeric composition as well as chain length between sites of crosslinks were varied to examine their effects on resin thermo-oxidative stability and glass transition temperature (Tg) of the cured/postcured resins. A linear correlation of decreasing 316 °C resin weight loss/surface area versus (a) decreasing aliphatic content or (b) increasing benzylic/aliphatic stoichiometry ratio over a wide range of resin compositions was observed. An almost linear correlation of Tg versus molecular distance between the crosslinks was also observed. An attempt was made to correlate Tg with initial composite mechanical properties (flexural strength and interlaminar shear strength). However, the scatter in mechanical strength data prevented obtaining a clear correlation. Instead, only a range of composite mechanical properties was obtained at 25, 288 and 316 °C. Perhaps more importantly, what did become apparent during the correlation study was (a) that PMR methodology could be used to prepare composites from resins that contain a wide variety of monomer modifications, (b) that these composites almost invariably provided satisfactory initial mechanical properties as long as the resins formulated exhibited satisfactory processing flow, and (c) that PMR resins exhibited predictable rates of 316 °C weight loss/surface area based on their benzylic/aliphatic stoichiometry ratio.			
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